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"A Study to Determine the Deformation Characteristics of Beryllium and Tungsten Under Conditions of High Hydrostatic Pressure"

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Prepared Under Navy, Bureau of Weapons
Contract No. N 600 (19) 59430

15 January through 14 March 1963

Interim Report No. 2

Pressure Technology Corporation of America
453 Amboy Avenue
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ABSTRACT

Tensile and compressive tests under pressure were performed on specimens of sintered polycrystalline tungsten and beryllium. Results are presented as reduction in area versus pressure. A brittle-ductile field is mapped for tungsten from available data.

Correlative fluid-extrusions were performed on sintered polycrystalline tungsten and beryllium, and on a single crystal of beryllium. There were indications that crack-free fluid-to-fluid extrusions should be possible for all materials, with additional modes of deformation apparently activated at higher pressure levels.

Analysis is presented on the role of pressure in plastic deformation, with extrusion to high-energy forming processes and to notch tensile testing.

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"A Study to Determine the Deformation Characteristics of Beryllium and Tungsten Under Conditions of High Hydrostatic Pressure"

I. Introduction

This report covers work performed during the period January 15, 1963 to March 14, 1963. Emphasis has been on the fundamental aspects of deformation under pressure, as obtained by determinations of physical properties in a pressurized environment. Both tensile and compressive tests were performed under pressure.

The determination of ductility as a function of pressure is believed to be a key to metalworking processes conducted without fracture at both ambient and elevated pressures. There are several aspects of the effect of pressure on ductility that are of interest. Among them are the variation with pressure of the fracture mode (brittle or ductile), of the extent of ductility, and of the critical resolved shear stress. There appears to be little existing theory on these phenomena.

A few correlative experiments were attempted with a single metalworking process that lent itself to use under pressure, namely, fluid extrusion.

This work was performed at Pressure Technology Corporation of America, Woodbridge, New Jersey.

II. Elementary Theory

A. General

The state of stress in a sufficiently small element of a solid can be represented completely by three principal stresses s_1, s_2, and s_3 (see figure 1), where compression is taken as positive. These stresses may have been generated by surface forces (pressure, friction, etc.) and/or by body forces (inertia, magnetic field, etc.).

The average direct stress, $\underline{s}$, is an invariant with respect to rotation of coordinate axes, and so may be found as the arithmetic mean of the principal stresses:

$$s = (s_1 + s_2 + s_3)/3.$$

The average direct stress is held to determine the change in volume of the element from that in an unstressed state, independently of the individual principal stresses, for materials that can be considered to be isotropic.

The principal-stress deviators are obtained by subtracting the average direct stress from the principal stresses:

$$s_1' = s_1 - s$$
$$s_2' = s_2 - s$$
$$s_3' = s_3 - s$$

Obviously, $s_1' + s_2' + s_3' = 0$.

Another invariant of rotation, the second deviatoric invariant, J_2', may be formed from the principal increases, and can be expressed in terms of the principal-stress deviators alone:

$$J_2' = s_1'^2 + s_2'^2 + s_3'^2.$$

For some materials, J_2' represents a criterion for plastic flow, employed as follows.

A uniaxial stress (tension or compression) is increased until yield occurs at stress Y. The principal stresses are Y, 0, 0. The average stress is Y/3. The principal-stress deviators are 2Y/3, -Y/3, -Y/3. The second deviatoric invariant is $2Y^2/3$. Whenever J_2' equals or exceeds $2Y^2/3$, plastic flow is held to occur.

B. Effect of Pressure on Criteria for Onset of Plastic Flow

The chief competitor to J_2' as a popular criterion for plastic flow is the maximum shear stress. As its name implies, plastic flow is held to occur when the maximum shear stress (in an element subjected to an arbitrary set of stresses) equals or exceeds the value of the maximum shear stress at which yield occurs in a specimen subjected to uniaxial stress.

Both criteria for plastic flow are held to be independent of <u>s</u>, at least to a first approximation. Most other theories of plastic flow are discarded because they include <u>s</u> as a factor influencing the onset of plastic flow.

The appeal of J_2' as a criterion of plastic flow is considerable:

a) it is a very simply expressed stress invariant;

b) it corresponds exactly to the octahedral shear-stress criterion that had been found independently;

c) it corresponds exactly to the elastic-distortion energy for an isotropic material;

d) it is independent of average direct stress;

e) it falls out directly and elegantly from the characteristic equation for deviator stresses; and

f) it agrees with experiment for some materials.

The maximum-shear-stress criterion, however, is not so different in its attributes, as follows:

a) it can be expressed in terms of deviatoric stress invariants (say, the second and third);

b) it is independent of average direct stress; and

c) it agrees with experiment for some materials.

It appears that a differentiation between the two criteria must be obtained from experimental instead of intellectual esthetic arguments. Such experimentation was conducted over a period of years on a few materials, with no great preponderance of support leaning towards either theory.

Relatively recently (reference 1), an appreciable amount of experimental data has been presented in English on the onset of plastic flow. One means of discriminating between the several criteria for plastic flow is the ratio of the shear stress at onset of flow in a torsion test, t , to the tensile stress at onset of flow in a tensile test, σ. The J_2' criterion predicts that $t = 0.578\ \sigma$, whereas the maximum-shear-stress criterion yields $t = 0.500\ \sigma$. Some of these data appear below.

Material	t/σ	Choice of Criterion For Onset of Flow
Iron, some steels, aluminum, copper	0.49-0.53	maximum shear stress
Some steels, an aluminum alloy	0.54-0.62	J_2'
Some steels, an aluminum alloy, a bronze	0.64-0.74	neither
Some aluminum alloys, magnesium, some magnesium alloys	0.25-0.41	neither

The conclusion is inescapable that further experimental investigation is required to furnish a firm basis for choice of a flow criterion for specific materials.

The flow criterion is important to metalworking processes in two ways:

a) it enables the yield stress (and strain-hardening characteristics) as determined in a tensile test to be employed in calculating forces required for specific deformation of a metal, and

b) it indicates the effect of average direct stress on forces required to deform metals.

One method of determining the effect of average direct stress on flow stress is fluid-to-fluid extrusion. The fluid pressure required to force a metal through a die into a lower fluid pressure can be determined. The difference in the two pressures characterizes the flow stress, and half the sum of the two pressures characterizes the average direct stress, to a first approximation. Such experimental data are already in existence for aluminum (reference 2a). It will be recalled that aluminum is one of the materials characterized by a flow criterion that is independent of pressure. The experimental data from reference 2 corroborate this independence, as follows (units of pressure, kg/cm^2):

% Reduction In Area	Half Sum of Extrusion And Receiving Pressures	Difference Between Extrusion and Receiving Pressures
44.5	625	1250
	3600	1200
	4650	1300
56.2	1000	2000
	1900	1800
	3325	1850
	4675	2050
75.4	1350	2700
	2550	2700
	3350	2700
	5000	2600

It may be concluded that the flow stress for aluminum is independent of average direct stress up to average direct stresses of at least 75,000 p.s.i., with experimental scatter in data of about plus or minus 5%-10%, for strains corresponding to area reductions up to 75%. This method lends itself to wider use, as for materials other than aluminum, for average pressures above 75,000 p.s.i., and for reductions other than 44%-75%.

C. Effect of Pressure on Brittle-Ductile Transition (BDT)

There has been presented elsewhere a brief review of experimental data on the effect of pressure on brittle-ductile transitions in solids (reference 3). For both metals and non-metals, a transition from brittle to ductile states was made possible by increase in pressure. A few materials, such as quartz, however, are resistant to the alteration from brittle to ductile states.

The BDT is well-known as a function of temperature, and less well-known in its dependence on pressure since fewer pressure than temperature data are available. The pressure characterizing a BDT will be termed the BDTP.

The BDTP is different when determined in tension, torsion, and compression. Let it be assumed, as a first approximation, that $\underline{P}$ at BDT is independent of stress state. Then the BDTP for torsion under pressure is $\underline{P}$, for tension is $(P + T/3)$, and for compression is $(P-C/3)$, where T and C are the yield stresses in uniaxial tension and compression respectively, and $\underline{P}$ is the environmental pressure.

This analysis is substantiated semi-quantitatively by data from tests under pressure, and qualitatively by the host of materials known to be brittle in tension and ductile in compression.

It thus appears that the BDTP determined by a series of tensile tests at different pressures is conservative for application to actual metalworking processes; the true BDTP will be lower than the pressure for the tensile BDT by about 1/3 the tensile stress at onset of flow.

D. Effect of Pressure on Ductility

Ductile materials gain in ductility with increasing pressure, in general. Thus, if a material cannot be worked a given amount without fracture, an adequate increase in environmental pressure will tend to permit fractureless deformation.

Ductility of some materials tends to "saturate" as pressure is increased; that is, pressure increase above a given pressure produces little further increase in ductility. Alpha brass is very well known for this behavior (reference 2b) in several kinds of experiments on deformation under pressure. For this reason, it isnot clearcut that pressure alone is the answer to increased ductility.

In many ways, pressure and temperature are interchangeable in effects on ductility. Pure materials that show sharp transitions with increasing temperature tend to have sharp values of BDTP. Less pure materials show gradual increases in ductility with rises in either temperature or pressure. In this regard, the pressure saturation of ductility of alpha brass is paralleled by its relative temperature independence of critical resolved shear stress.

Both the BDT and increase of ductility with pressure indicate that increase in pressure can activate additional modes of deformation, just as does temperature. This behavior is of interest for materials that are difficult to deform at atmospheric pressure.

E. <u>High-Energy Deformation (HED)</u>

The preceeding discussion appears to have bearing on high-energy deformation processes.

It is well known that an increase in the rate of tensile straining yields a decrease in ductility for both single crystals and polycrystals. Nevertheless, explosive forming, magnetic forming, etc., are all high-strain-rate processes that permit larger deformations than do similar static processes. These successful rapid-straining processes have in common a high-pressure environment generated by body forces, chiefly inertia.

It consequently appears that the success of high-energy deformation is attributable to the pressure environment and not to the rapid straining <u>per se</u>. In fact, the rapid straining tends to reduce ductility.

The same results obtained by HED should be obtainable by static deformation under pressures equal to those generated by the HED. In fact, better results should be available from static processes since the pressure acting on the specimen material does not vary from point to point.

It is thus indicated that attention given to increasing the level and uniformity of inertially-generated pressure can yield better controlled properties in materials subjected to HED.

F. Notch Testing

Notched specimens subjected to uniaxial tension appear more brittle than do unnotched specimens. It is well known that a state of triaxial tension is produced in such a notch tensile test. The average direct stress in a notch tensile test is tensile, tending to reduce the ductility of the material.

In principle, the superposition of an environmental pressure equal to the average direct tensile stress in the notched specimen should restore a ductility equal to that of the unnotched specimen, in the absence of large stress-concentration effects.

It thus appears that a BDTP should be characteristic of a given material with a given notch geometry, in tensile test. Tensile tests under pressure should consequently be a sensitive criterion of the alteration in notch brittleness of a material as its grain size, heat treatment, etc., are varied.

Tensile tests under pressure should also be capable of assigning BDTP's to notches of different geometries, thus tending to correlate results of notch tensile tests from different shapes (and sizes) of specimens.

III. Tensile Tests Under Pressure

A. First Tensile Fixtures

Tensile tests were conducted on dummy specimens with the fixtures described in the previous progress report. With specimens of both aluminum and steel, the same difficulties arose.

The button heads of a specimen, although supported by the ends of the fixtures through an angle greater than 180°, tended to translate away from the centerline of the fixtures because the center of force on the fixtures did not coincide with the centerline of the specimen. Further, the use of large fillets on the tensile specimens resulted in small bearing areas on the heads of the specimens. The results of these factors were bending of the specimens and bearing failure on the heads. Several minor modifications were made of the design of the fixtures, but the improvements in performance were marginal.

B. Final Tensile Fixtures

An older design of tensile fixture was next adopted that had the advantages of coincidence of center of force on the fixture and centroid of the specimen, and increased bearing area of fixtures on button heads of tensile specimens.

The minor disadvantages of these fixtures were that assembly was more complex than with the first fixtures, and that sixteen components comprised the new tensile-test assembly compared to the previous two-piece assembly.

Tensile tests on dummy specimens were now successful. A dead-brittle material was pulled under no pressure, with the usual brittle failure. Under pressure, necking could be obtained, as shown in figure 2.

Tensile tests were next conducted on sintered polycrystalline tungsten and on sintered polycrystalline beryllium.

Results of tensile tests on sintered polycrystalline tungsten are given in figure 3. An approximate brittle-ductile demarcation line is shown rising from about 90,000-100,000 p.s.i. with zero per cent reduction in area after test, to about 250,000 p.s.i. with 50% reduction in area.

Three of the specimens are shown after test, and one before test, in figures 4 and 5. The fractured surfaces of the tungsten specimens are all "brittle", manifesting no cup-cone behavior.

Results of tensile tests on sintered polycrystalline beryllium are not yet adequate for definite interpretation. One test at 246,000 p.s.i. resulted in a brittle fracture at the fillet ending the gage length. Inspection revealed gross machining marks at the fillets that probably localized failure. In an effort to duplicate the test, another specimen was well polished both at the fillets and along the entire gage length. It failed in tension by a gradual pulling apart at extremely low stress under a pressure not exceeding 229,000 p.s.i. The failure was localized at the center of the gage length, but reduction in area was small, less than 4.9%.

Determination of stresses in tensile specimens will be made from the point-by-point load-deflection data available for each test, after elastic calibrations are made of the tensile fixtures and supports.

The unexpectedly great ductility under pressure of the tungsten specimens requires that tensile fixtures of greater extension be supplied, to permit tests to be carried to fracture at higher pressure. Such fixtures were designed and fabricated.

IV. <u>Compression Tests</u>

Compression tests under pressure have been made of both sintered polycrystalline tungsten and beryllium specimens.

The specimen was shaped as a cylinder of equal height and diameter, shown in figure 6.

Results for beryllium are shown in part in figure 6. Specimens compressed 10% and 12% respectively under pressures not exceeding 190,000 p.s.i. showed no cracking (not illustrated). A specimen compressed about 60% in length at atmospheric pressure (under stress of about 300,000 p.s.i.) showed more edge cracking than did another specimen compressed about the same amount under pressure of approximately 150,000 p.s.i.

Results for tungsten are incomplete. A preliminary run yielding about a 10% change in length under pressure of about 125,000 p.s.i. showed no failure. At atmospheric pressure, a specimen deformed 40% in length showed edge cracking.

V. Use of Fluid-To-Fluid Extrusion As A Criterion For Deformation Under Pressure

A small investigation was made of fluid-to-fluid extrusion as a tool to correlate more fundamental theory and data with actual metalworking under pressure.

A beryllium single crystal was kindly furnished by Franklin Institute for such extrusion tests. Unfortunately, no tensile nor compressive specimens of single crystal beryllium are available for better correlation. The partially-extruded specimen is shown in two views in figure 7. The small-diameter nose was present on the original as-machined specimen. The partial extrusion is the middle cylindrical portion. A small amount of dragging on the die wall took place resulting in the slight bending shown in one view. In general, the conclusion is that deformation is reasonably isotropic. The possible explanation is that additional modes of deformation were made available by the 300,000 p.s.i. pressure level at which the extrusion was performed.

Since this is the only single crystal of Be presently available, an attempt is being made to cut other specimens from it, for compressive test. A first attempt by gentle sawing with a jeweler's saw proved abortive and produced some cracking. A next attempt will be to cut the specimen by repeated rubbing with a moving acid-soaked filament.

Extrusions of sintered polycrystalline beryllium were also attempted. A 10% reduction completely fluid-extruded into zero pressure by an extrusion pressure of 135,000 p.s.i. resulted in a finely cracked extrusion in many pieces. A 20% reduction partially fluid-extruded into a pressure of about 100,000 p.s.i. is shown as the longer extrusion in figure 8. It can be seen that, although cracks are present, the extrusion is still integral. Another 20% reduction partially fluid-extruded into a pressure of about 150,000-200,000 p.s.i. produced the short uncracked piece shown in figure 8.

Extrusion of sintered polycrystalline tungsten was briefly examined. Some results are shown in figure 9. The longest specimen was reduced 20% by fluid-extrusion into zero pressure, yielding a cracked and broken result. A smaller reduction was achieved, crack-free, as shown in the middle specimen of figure 8. The lower specimen manifests two partial extrusions. A 20% reduction into a pressure of 150,000-200,000 p.s.i. yielded a slightly cracked product. The same billet was further extruded into the same pressure with a 14% reduction, resulting in a crack-free product.

These W extrusions, although some were crack-free, are not thought to be typical of what can be accomplished with this material. There is a large suspicion that the as-received and slightly cleaned surface of the billets contains small cracks, probably longitudinal in disposition. Not shown in any illustrations are several extrusions that possessed longitudinal cracks after partial extrusion, on portions that had not been reduced! Several samples of W rod have consequently been centerless-ground to remove .005" from the surface, in anticipation of further experimentation with more nearly crack-free material.

In general, there is also some suspicion that liquid under pressure, in contact with porous materials (such as these sintered metals) can reduce ductility. This embrittling effect has been noted for cast iron and some magnesium alloys, at least, by other investigators.

It is consequently thought desirable to conduct a few tensile tests under pressure of these materials while covered by an elastomeric adhesive to prevent contact with the fluid under pressure.

VI. Future Work

Tensile tests will be continued of W and Be materials, and compressive tests of W single crystals. Compression of a Be single X-tal will be attempted if this specimen can be obtained. The compression tests of the W single crystal will be done in the directions of at least two different axes.

Tensile tests will be examined for Be and W specimens not in contact with fluid under pressure.

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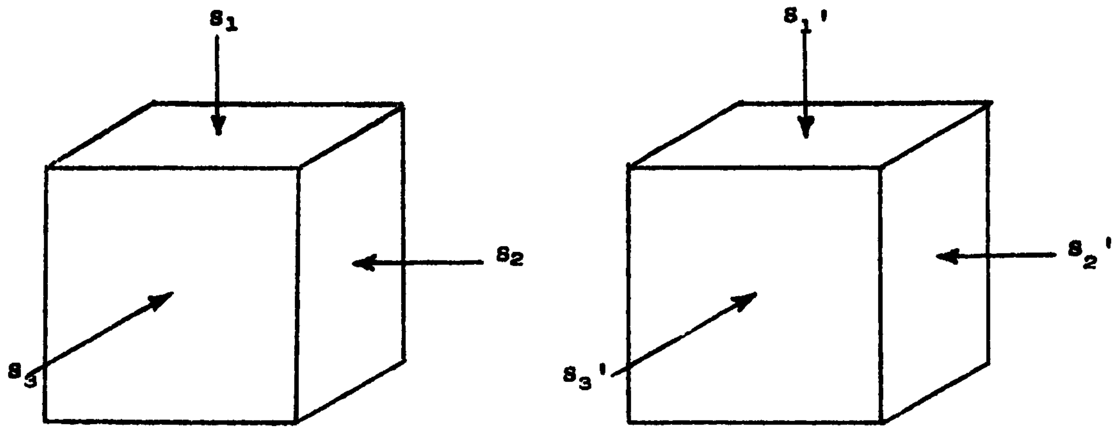
1. UNKSOV, E.P.: "An Engineering Theory of Plasticity", translated to English, Butterworths, London, 1961, (original published Moscow, 1959), Chapter 5.

2.a. BERESNEV, B.I.; VERESHCHAGIN, L.F.; RYABININ, Yu.N.; and LIVSHITS, L.D.: "Some Problems of Great Plastic Deformation in Metals Under High Pressures", Akad.Nauk, Moscow, 196C, Table 8.

2.b. Ibid, Figure 5.

3. BOBROWSKY, A.; and STACK, E.A.: "Deformation of Metals Under High Pressure", presented at the Dallas meeting of AIME, February 1963, to be published as part of symposium proceedings.



Principal stresses	s_1 , s_2 , s_3
Average direct stress	$s = (s_1 + s_2 + s_3)/3$
Principal-stress deviators	$s_1' = s_1 - s$ $s_2' = s_2 - s$ $s_3' = s_3 - s$

$\therefore s_1' + s_2' + s_3' = 0$

Sign convention: Compressive stresses are positive.

Figure 1: Nomenclature for stresses.

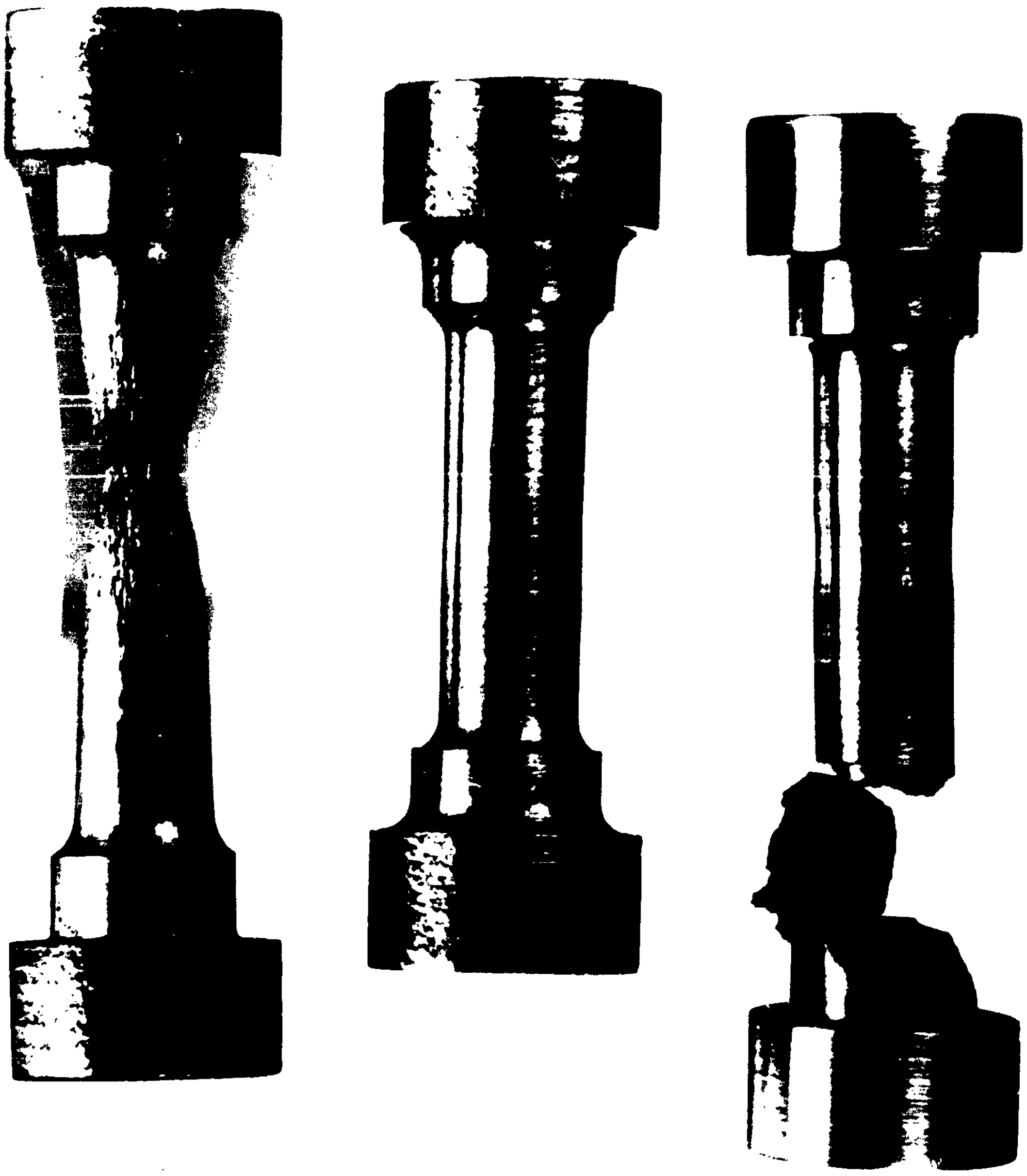


Figure 2: Tensile Tests Under Pressure.
Left to right: Ductile failure of normally brittle dummy material, original specimen, brittle failure of beryllium specimen.

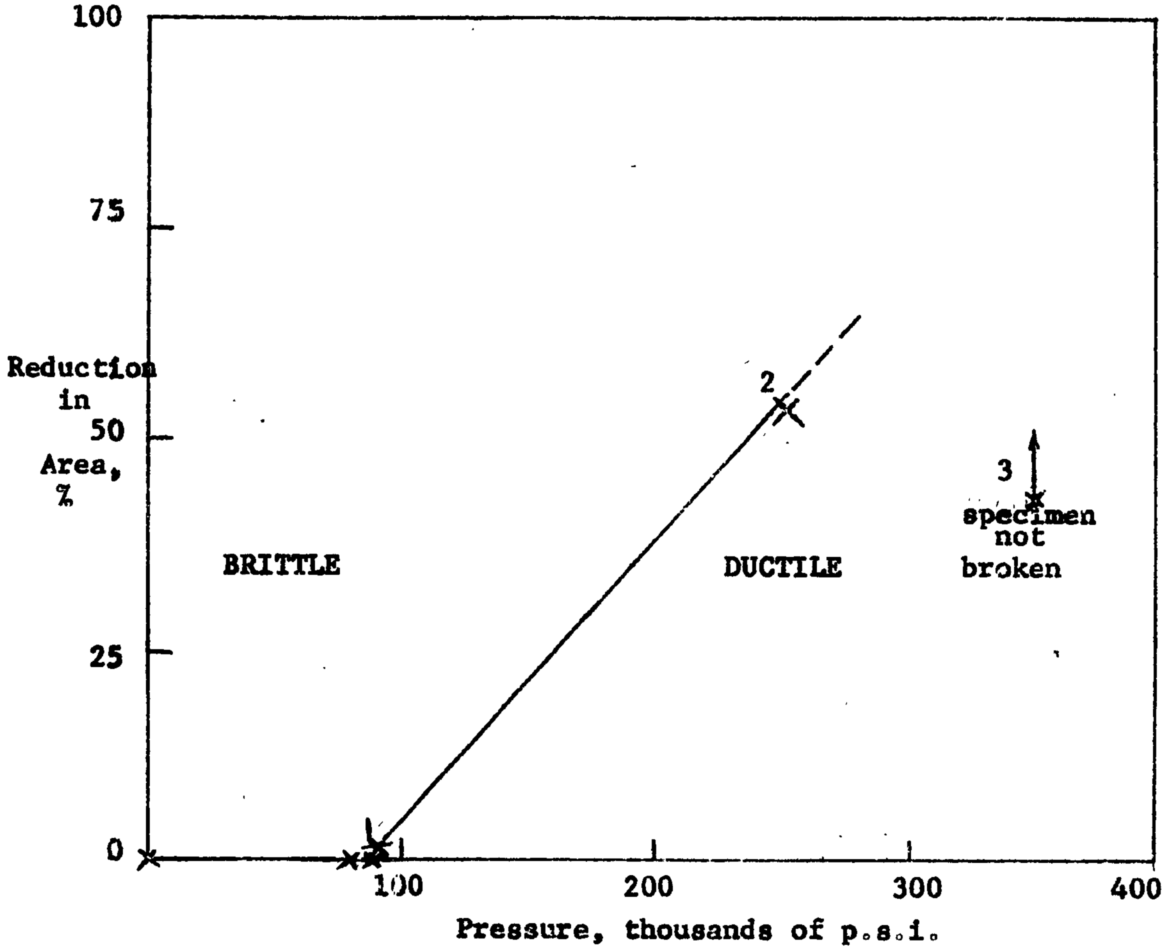


Figure 3: Results on Tensile Tests Under Pressure for Polycrystalline Tungsten, Surface As Received. Specimen Nos. 1 and 2 are shown in figure 4; specimen No. 3 and a specimen before test are shown in figure 5.

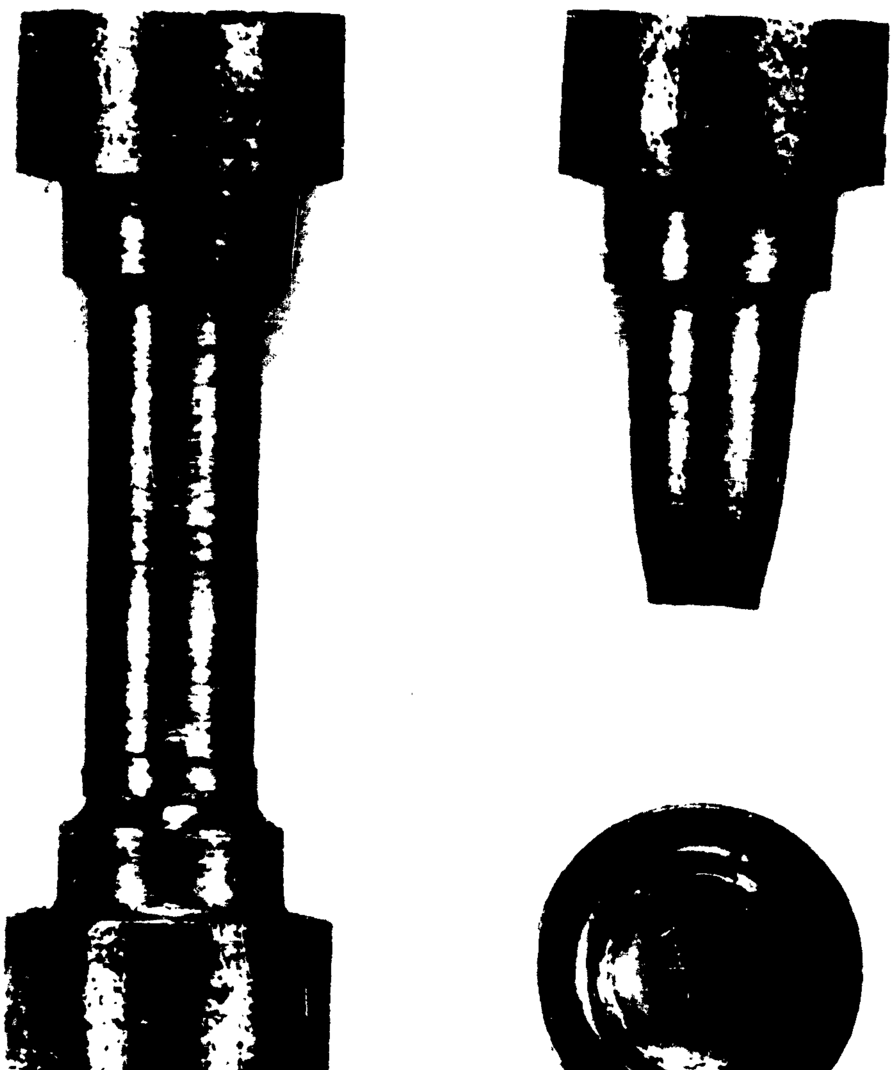


Figure 4: Tensile Tests Under Pressure.
Left to right: Brittle fracture of polycrystalline tungsten under pressure of 90,000 p.s.i.; ductile behavior of polycrystalline tungsten under pressure of 253,000 p.s.i.

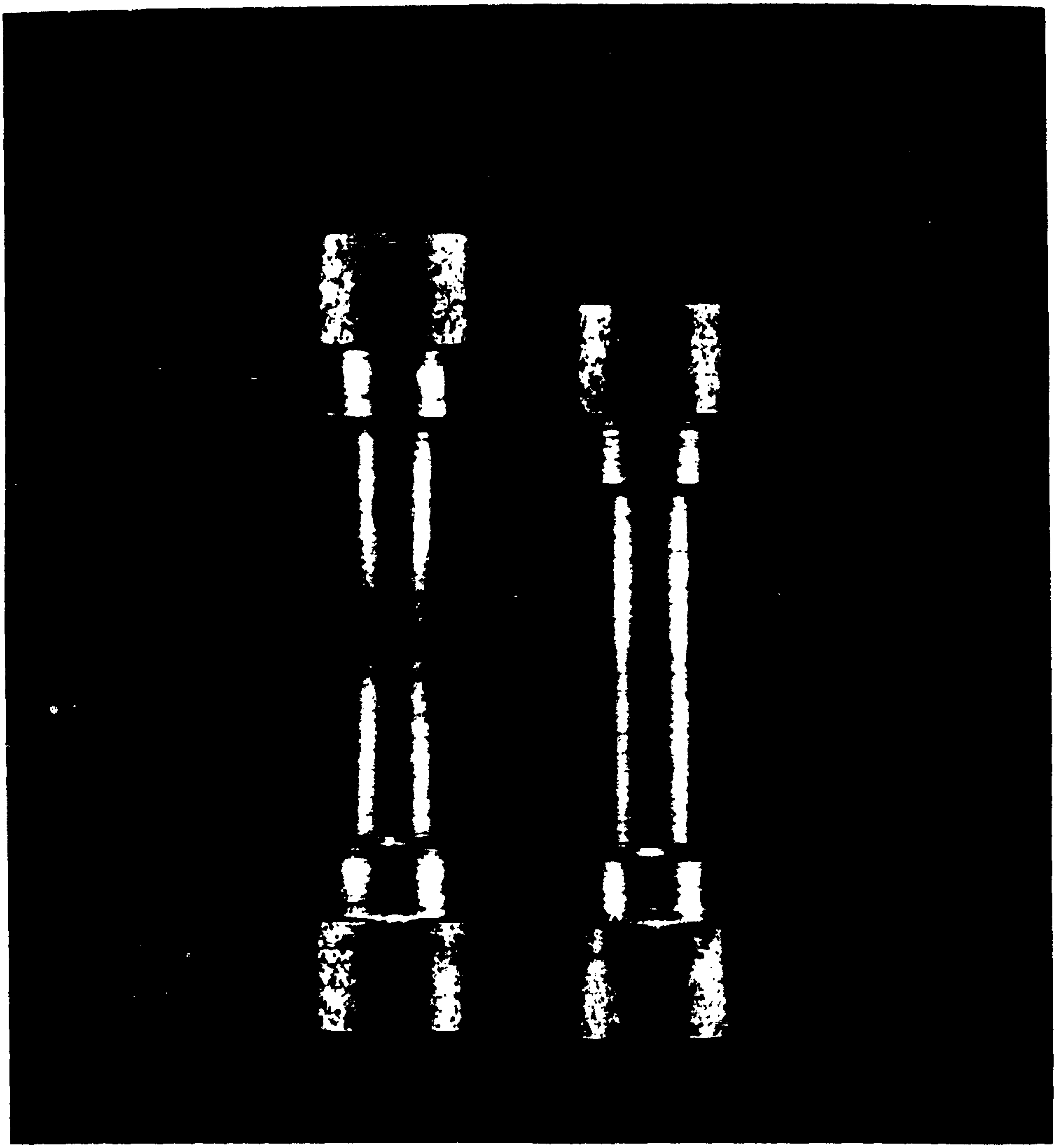


Figure 5: Tensile Tests Under Pressure.
Left to right: Ductile behavior of polycrystalline tungsten under pressure of 350,000 p.s.i. with test stopped because of limitation in extension by tensile fixture; polycrystalline specimen of tungsten before test.

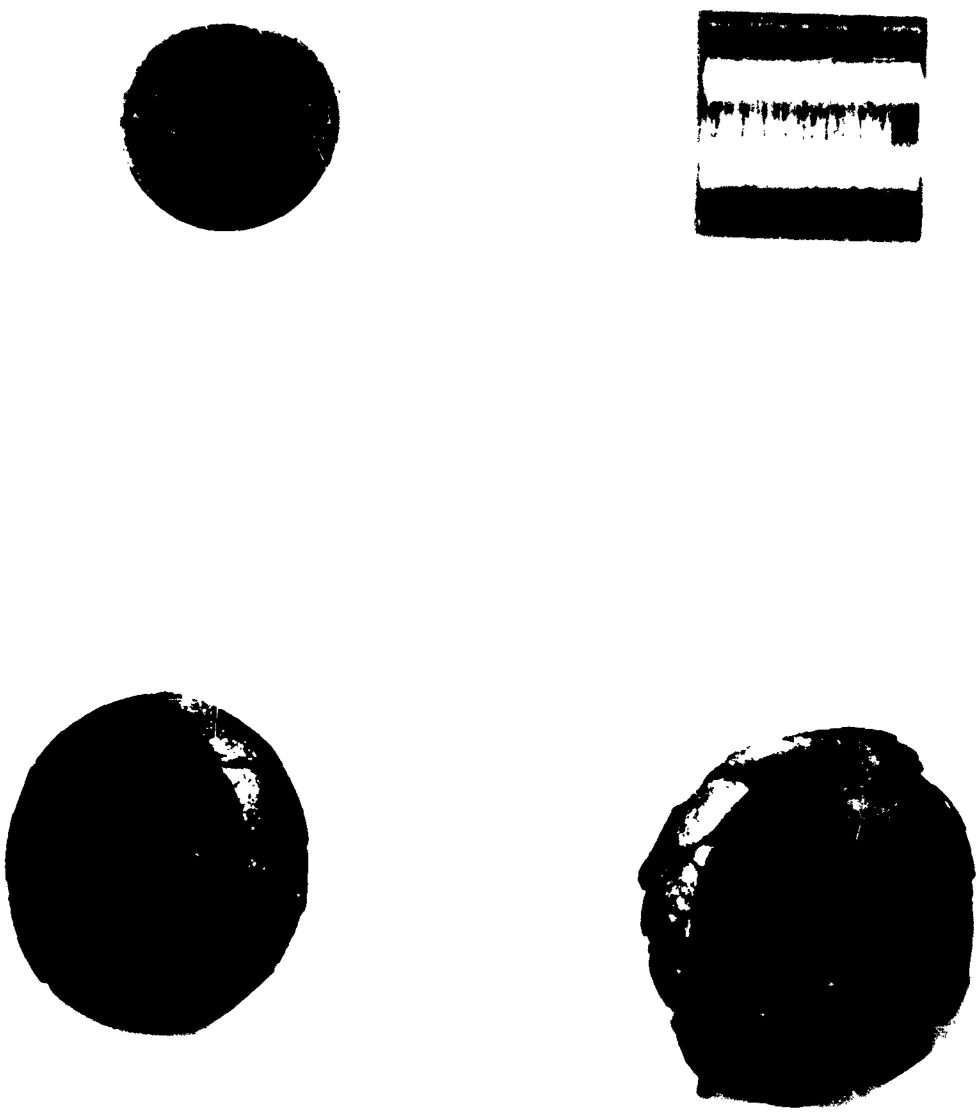


Figure 6: Compression Tests.
Top: Specimen before test, two views.
Bottom: Left, beryllium under pressure;
Right, beryllium with no pressure.

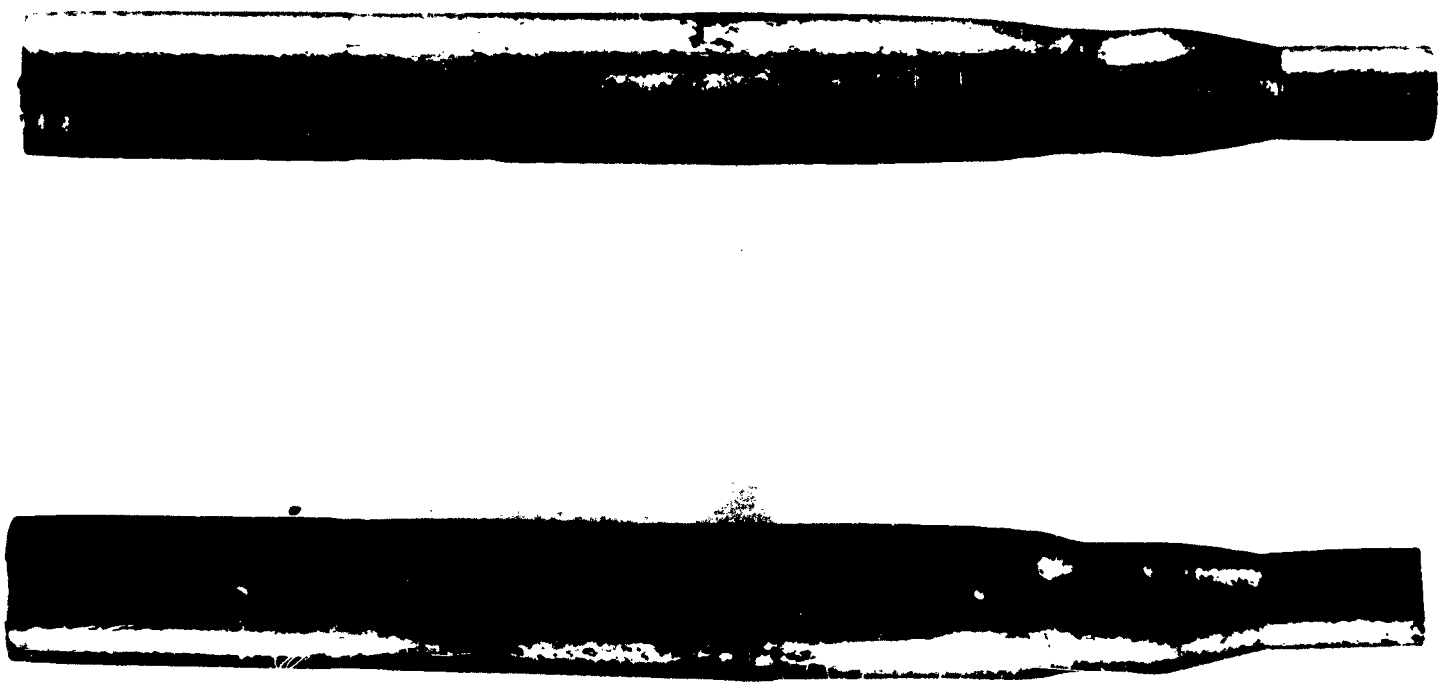


Figure 7: Beryllium single crystal, partially fluid-extruded, showing ability to be worked without excessive anisotropic deformation

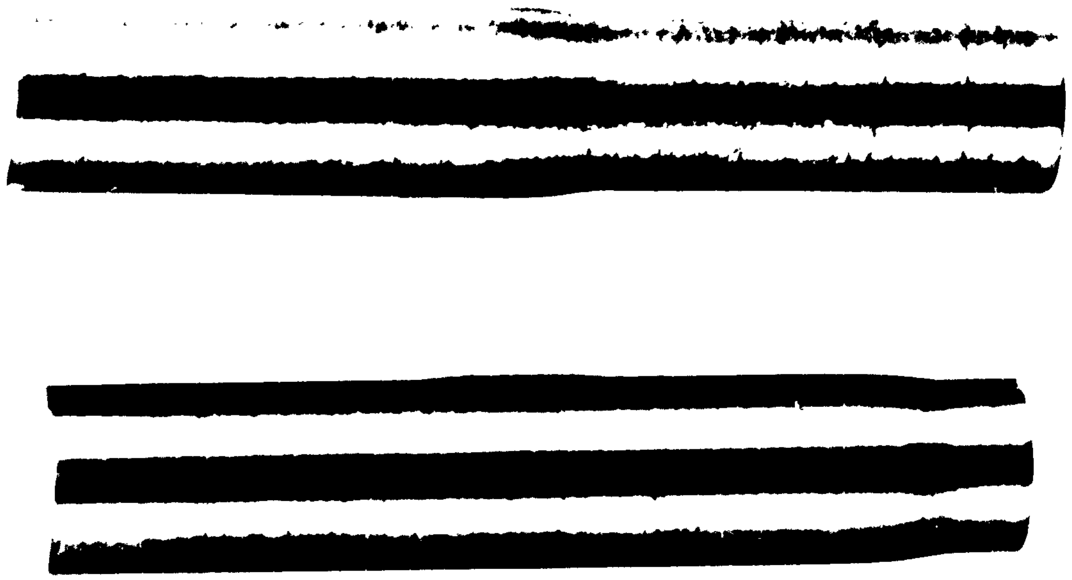


Figure 8: Fluid-To-Fluid Extrusions.
Sintered polycrystalline beryllium, shorter uncracked extrusion performed into higher pressure than longer cracked extrusion.

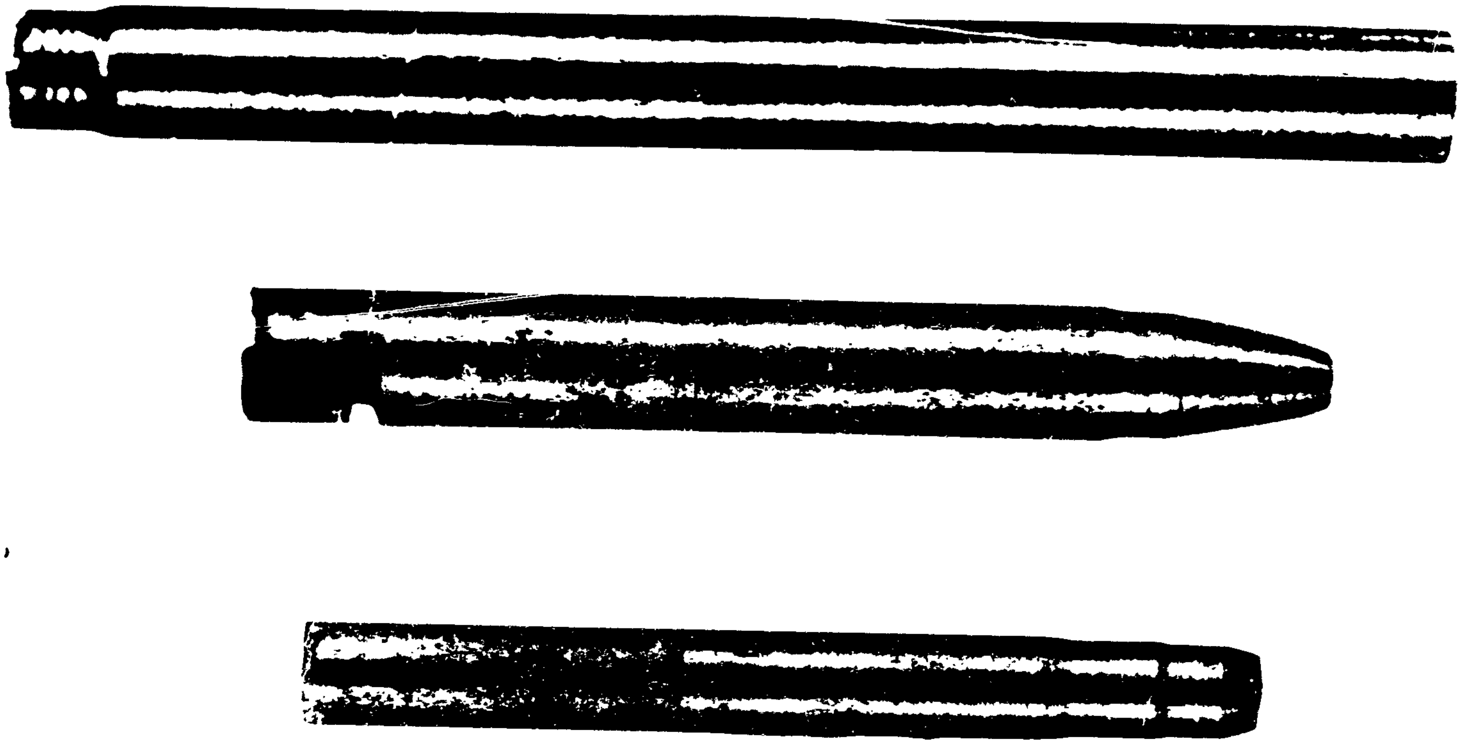


Figure 9: Fluid-to-fluid extrusions of tungsten.